भारतीय मानक
Indian Standard

IS 6831 : 2023

कास्टिक पोटाश — विशिष्टि

(तीसरा पुनरीक्षण)

Caustic Potash — Specification

(Third Revision)

ICS 71.060.50

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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1973 and revised in 1982 and 1992. It was revised in 1982 changing the technical grade as pure grade as the caustic potash manufactured by electrolytic process is of high purity. Also, changes were made in the requirements of chlorides, sulphates, iron, heavy metals, silicates and nickel for pure and AR grades. Since caustic potash is also manufactured in substantial quantity in lye form, it was included in the first revision. Some modifications were also made in the preparation of sample solution and methods for determination of total alkalinity, carbonates, chlorides and sulphates. In the second revision, a separate grade of caustic potash suitable for use in electronic industry was incorporated. An alternative method for chloride determination was also incorporated.

In this revision, instrumental test methods for the determination of aluminium, arsenic, calcium, chlorides, iron, lead, magnesium, nickel, nitrates, sodium, sulphates and zinc have been added as alternate test methods. Also, Packing and Marking clause and Reference clause have been updated. Further, Amendment has been incorporated in this revision.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022. ‘Rules for rounding off numerical values (*second revision*)’.The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CAUSTIC POTASH — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for caustic potash.

1.1.1 It covers the material in solid and lye forms.

2 REFERENCES

The following standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
IS 2088 : 1983	Methods for determination of arsenic (*second revision*)
IS 3025 (Part 2) : 2019/ISO 11885 : 2007	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (*first revision*)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)

3 GRADES

There shall be three grades of the material as follows:

a) *Pure* — suitable for use in the manufacture of soaps, oxalic acid and other potassium compounds. It may also be used for process engraving and lithography and for manufacture of products like dyestuff and potassium permanganate;

b) *Analytical Reagent* (*AR*) — suitable for use as reagent in analytical work; and

c) *Electronic Grade* (*ER*) — suitable for use in electronic industries.

4 REQUIREMENTS

4.1 Description

The solid material shall be in the form of pellets or flakes having a crystalline structure. It shall be free from dirt, foreign matter and other visible impurities. In case of lye, the material shall be clear and transparent liquid, free from visible suspended impurities.

4.2 The material shall comply with the require merits given in Table 1 when tested according to methods prescribed in Annex A. Reference to relevant clauses of Annex A is given in co1 (6) of Table 1.

4.3 Optional Requirements for Pure Grade

If required by the purchaser, the limit for arsenic shall be as agreed to between the purchaser and the supplier.

5 PACKING AND MARKING

5.1 Packing

The pure grade of the material shall be packed in high density polyethylene or polypropylene bags or in mild steel drums lined with low density polyethylene or as agreed to between the manufacturer and supplier. The analytical reagent grade and electronic grade material shall be packed in glass bottles or as agreed to between the

manufacturer and supplier.

5.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Name of the manufacturer and his recognized trade mark, if any;
c) Gross and net mass;
d) Date of manufacture; and
e) Batch number.

5.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Caustic Potash
(*Clause* 4.2 *and* 5.2.1.2)

Sl No.	Characteristic	Requirement			Methods of Test, Ref to
(1)	(2)	(3)	(4)	(5)	(6)
i)	*Total alkalinity (as KOH), percent by mass, *Min*	87.0	87.0	87.0	**A-3**
ii)	Carbonates (as K_2CO_3), percent by mass, *Max*	1.0	1.0	1.0	**A-4**
iii)	Chlorides (as KCl), ppm, *Max*	300.0	40.0	40.0	**A-5** or **A-21**
iv)	Sulphates (as K_2SO_4), ppm, *Max*	50.0	50.0	20.0	**A-6** or **A-21**
v)	Phosphates (as PO_4), percent by mass, *Max*	—	0.000 5	0.000 5	**A-7**
vi)	Iron (as Fe), ppm, *Max*	25.0	5.0	5.0	**A-8** or **A-20**
vii)	Heavy metals (as Pb), ppm, *Max*	10.0	5.0	5.0	**A-9** or **A-20**
viii)	Insoluble matter, percent by mass, *Max*	—	0.01	0.005	**A-10**
ix)	Sodium (as Na), percent by mass, *Max*	1.0	0.5	0.05	**A-11** or **A-20**
x)	Arsenic (as As), percent by mass, *Max*	—	0.000 1	0.000 1	**A-12** or **A-20**
xi)	Nitrates (as NO_2), percent by mass, *Max*	—	0.001	—	**A-13 or A-21**
xii)	Silicates (as SiO_2) ppm, *Max*	200.0	50.0	40.0	**A-14**
xiii)	Aluminium (as Al), percent by mass, *Max*	—	0.002	0.001	**A-15** or **A-20**
xiv)	Ammonium salts (as NH_4), percent by mass, *Max*	—	0.000 5	—	**A-16**
xv)	Calcium and magnesium (as Ca), percent by mass, *Max*	—	0.002 5	0.002	**A-17** or **A-20**

Table 1 (*Concluded*)

Sl No.	Characteristic	Requirement			Methods of Test, Ref to
(1)	(2)	(3)	(4)	(5)	(6)
xvi)	Nickel (as Ni), ppm, *Max*	20.0	10.0	—	**A-18** or **A-20**
xvii)	Zinc (as Zn), percent by mass, *Max*	—	0.001 5	—	**A-19** or **A-20**

*In case of the material in lye form, the total alkalinity (as KOH) shall be 45 percent to 50 percent by mass. The limits prescribed for other characteristics shall not exceed when calculated to a total alkalinity of 87.0 percent by mass.

5.2.1.2 In the case of analytical reagent grade, the maximum limits for the characteristics given in co1 (4) of Table 1 shall also be given in the label.

5.2.1.3 The containers may also be marked with the Standard Mark. The details may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The procedure for drawing the representative samples of the material and their criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(*Clause* 4.2 *and Table* 1)

METHODS OF TEST FOR CAUSTIC POTASH

A-1 QUALITY OF REAGENTS

A-1.1 unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in a tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 PREPARED SAMPLE SOLUTION

A-2.1 In a weighing bottle with a ground-glass stopper, weigh the material (solid or lye), to the nearest 0.01 g equivalent to a little less than 50 g of caustic potash.

A-2.1.1 *Solid Material*

Dissolve the material (**A-2.1**) in approximately 200 ml of water and cool it to room temperature. Transfer the solution to a 500 ml one mark volumetric flask and dilute nearly to the mark. Cool again to the room temperature, dilute to the mark and mix thoroughly. The solution thus prepared shall be used for the subsequent tests.

A-2.1.2 *Liquid Material*

Transfer the material (*see* **A-2.1**) directly to a 500 ml one mark volumetric flask and dilute with water. Cool to room temperature, dilute to the mark and mix thoroughly. The solution thus prepared shall be used for tests.

NOTE — Caustic potash rapidly deliquesces. It is also caustic lo organic matter. It should be kept in a tightly closed container and handled with rubber gloves on.

A-3 DETERMINATION OF TOTAL ALKALINITY

A-3.1 Reagents

A-3.1.1 *Standard Hydrochloric Acid* — 1 N

A-3.1.2 *Methyl Orange Indicator*

Dissolve 0.1 g methyl orange in 100 ml of water.

A-3.2 Procedure

Transfer exactly 20 ml of the prepared sample solution (*see* **A-2)** to a 500 ml conical flask. Add approximately 80 ml of water and 5 drops of methyl orange indicator and titrate against standard hydrochloric acid until the colour of the indicator changes from yellow to orange.

A-3.3 Calculation

Total alkalinity (as KOH), percent by mass

$$= \frac{V \times \mathrm{N}}{M} \times 140$$

where

V = volume of standard hydrochloric acid used in titration, in ml;
N = normality of standard hydrochloric acid used for titration; and
M = mass of the material taken for the test, in g.

A-4 DETERMINATION OF CARBONATES

A-4.1 Two methods, namely, Method A ‘Double Indicator Method’ and Method B ‘Gaseometric Method’ have been prescribed. Method A shall be used for routine analysis and Method B shall be used as a referee method.

A-4.2 Double Indicator Method — Method A

A-4.2.1 *Reagents*

A-4.2.1.1 *Standard hydrochloric acid* **—** 1 N and 0.1 N

A-4.2.1.2 *Methyl orange indicator*

Dissolve 0.1 g of methyl orange in 100 ml of water.

A-4.2.1.3 *Phenolphthalein indicator solution*

Dissolve 0.1 g of phenolphthalein powder in 60 ml of rectified spirit (*see* IS 323) and dilute with water to 100 ml.

A-4.2.2 Procedure

Pipette out 25 ml of the prepared sample solution (*see* **A-2**) into a conical flask and add 2 to 3 drops of phenolphthalein indicator. Titrate it against standard hydrochloric acid solution (1 N) up to a little before the end point. Take this reading as A. Further titrate it against standard hydrochloric acid (0.1 N) till the pink colour just disappears. Take this reading as B. Then add 2 to 3 drops of methyl orange indicator and continue titration against standard hydrochloric acid (0.1 N) to a reddish orange colour. Take this reading

as C.

A-4.2.3 Calculation

Carbonates (as K_2CO_3), percent by mass

$$= \frac{276(C - B) \times N}{M}$$

where

N = normality of standard hydrochloric acid (0.1 N); and

M = mass, in g, of the material in the aliquot taken for this test.

NOTE — The reading A which is taken with 1 N hydrochloric acid will be converted to the equivalent volume of 0.1 N hydrochloric acid before calculating volumes *B* and *C*.

A-4.3 Gaseometric Method — Method B

A-4.3.1 *Principle*

The volume of carbon dioxide evolved from a portion of the test sample by reaction with dilute hydrochloric acid is measured.

A-4.3.2 *Reagents*

A-4.3.2.1 Distilled water, or water of equivalent purity, free from carbon dioxide at room temperature. Eliminate any carbon dioxide present either by boiling water for 10 min and cooling it in the absence of atmospheric carbon dioxide, or, more simply, by bubbling air free from carbon dioxide through it for 15 min. The air is freed from carbon dioxide by passing it through a column containing pellets of sodium hydroxide. Store the water in an atmosphere free from the carbon dioxide.

A-4.3.2.2 *Hydrochloric acid* **—** 1 N

A-4.3.2.3 *Sodium chloride, coloured acid solution*

Dissolve 26 g of sodium chloride, in water. Add 5 ml of sulphuric acid (relative density = 1.83. Dilute it to 1 000 ml, add a few drops of 0.05 percent methyl orange indicator solution and mix thoroughly.

A-4.3.2.4 *Sodium hydroxide solution* **—** 1 N

A-4.3.3 *Apparatus* **—** as shown in Fig. 1

A-4.4 Procedure

A-4.4.1 Weigh, to the nearest 0.01 g, the test sample (solid or lye) corresponding to approximately 4 g to 5 g of caustic potash.

A-4.4.2 *Preparation of the Apparatus*

Fill burette B of the apparatus with the coloured acid solution through levelling bottle F. Pour into absorber C some of the potassium hydroxide solution (renew this solution after 100 determinations). With burette B and absorber C filled up to stop cock R, and graduation mark a respectively, and stop cocks R, and R, closed, place the test portion in flask A (in the case of solid material, dissolve it in approximately 30 ml of water). Dilute to approximately 40 ml so as to reduce the dead space to a volume slightly greater than 100 ml (volume above the level of the liquid in flask A plus the volume of condenser tube D up to cock R,). Place in the flask three porcelain or glass balls, about 1 mm in diameter, and a few pieces of pumice having a total volume approximately equal to that of the balls. Stopper the flask and close the stop cock R. Connect flask A with burette B through the stop cock R, and lower the levelling bottle F. Check the tightness of the apparatus by appropriately handling the cocks and the levelling bottle.

A-4.4.3 *Evolution and Measurement of Carbon Dioxide*

By means of a separating funnel, pour 35 ml of 12 N hydrochloric acid into flask A taking care to avoid loss of gas. The acidity of the solution in the flask is thus approximately 2 N after the evolution of carbon dioxide. Heat the flask and maintain the solution at boiling point for 5 min while running cold water through the condenser. Then stop heating and add more of the coloured acid solution by means of the separating funnel, lowering the levelling bottle F still further to make the solution in the flask A rise in the condenser tube up to cock R_1. Then close the latter and wait for 5 min to allow the gas to reach the temperature of the water jacket. Measure the volume of gas vat atmospheric pressure P and at the temperature t of water in the jacket. For this purpose move the levelling bottle F to bring the coloured acid solution to the same level both in the flask and in the burette B; read the volume of the latter. Adjust the stop cocks R_1 and R_2 so that the burette B and absorber C are connected, then raise the levelling bottle F so that the gas is transferred to the absorber where the carbon dioxide is absorbed. Then transfer the non-absorbed gas back to burette B by lowering the levelling bottle F and, after adjusting the level of the hydroxide solution to the gauge mark a on absorber C, close stop cock R_2 and read the volume of the residual gas. Repeat these operations until a obtained constant volume V_1 is The difference in the volume ($V - V_1$) represents the carbon dioxide contained in the test portion, measured at atmospheric pressure and at the temperature of the water jacket.

A-4.4.4 Calculation

Carbonates (as K_2CO_3), percent by mass

$$= 0.222\,7\ \frac{P - p}{273 + t} \times \frac{V - V_1}{M}$$

where

P = atmospheric pressure, in mm Hg, at the time of determination;

p = vapor pressure, in mm Hg, of the coloured acid solution at the temperature of the water in the jacket;

t = temperature, in degrees celsius, of the water in the jacket;

V = volume, in ml, of gas before absorption of carbon dioxide;

V_1 = volume, in ml, of gas after absorption of carbon dioxide; and

M = mass, in g, of the material taken for the test.

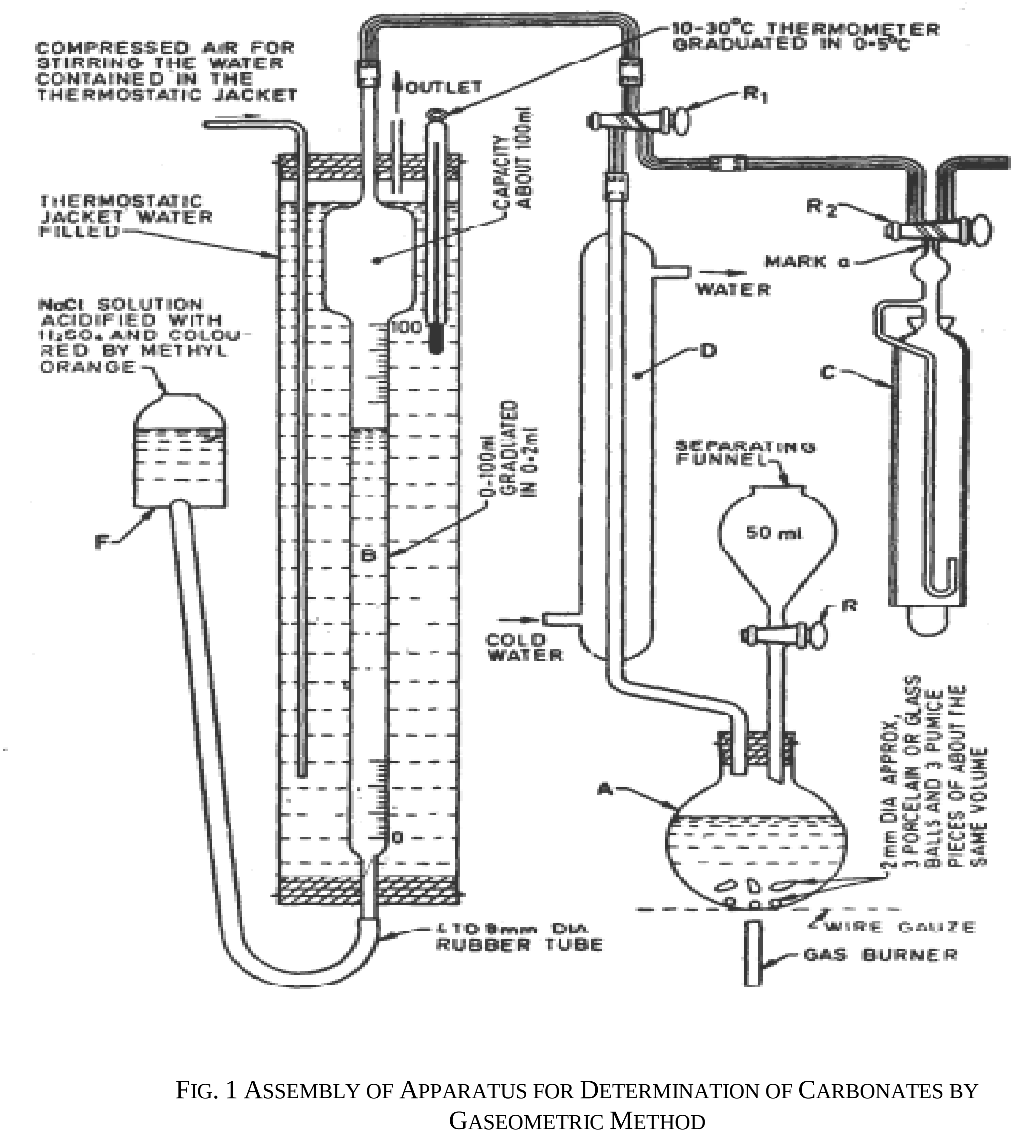

FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF CARBONATES BY GASEOMETRIC METHOD

A-5 TEST FOR CHLORIDES

A-5.1 Two methods, namely, Method A, Turbidity Method and Method B Volumetric Method have been prescribed. In case of dispute the Turbidity Method shall be the referee method.

A-5.2 Turbidity Method — Method A

A-5.2.1 *Apparatus*

A-5.2.1.1 *Nessler cylinders* **—** 100 ml capacity

A-5.2.2 *Reagents*

A-5.2.2.1 *Dilute nitric acid* **—** approximately 5 N

A-5.2.2.2 *Silver nitrate solution* **—** approximately 5 percent (*m/v*)

A-5.2.2.3 *Standard chloride solution*

Dissolve 0.210 g of potassium chloride (dried at 105 ± 2 °C) in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of chloride as Cl or 0.21 mg as KCl.

A-5.2.3 *Procedure*

Weigh accurately 5 g of the material for pure grade and 10.0 g of the material for AR and ER grades into a Nessler cylinder. Dissolve in 30 ml of water (chloride-free). Add 20 ml of dilute nitric acid for pure grade and 40 ml dilute nitric acid for analytical grade followed by 1 ml of silver nitrate solution. Dilute to the mark. Carry out a control test in another Nessler cylinder using 7 ml standard chloride solution for the pure grade and 2 ml standard chloride solution for the AR and ER grades in place of the material and the same quantities of the other reagents in the same total volume of the reaction mixture. Compare the turbidities produced in the two cylinders after 5 min.

A-5.2.3.1 The limit prescribed for chlorides shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test. **A-5.3** Volumetric Method - Method B. **A-5.3.1** Reagents **A-5.3.1.1** Silver nitrate solution - 0.01 N. Weigh accurately 0.169 9 g of silver nitrate (AR grade) and dissolve in chloride free water. Add 10 ml of concentrated nitric acid and dilute to one litre with water. Store the solution in a coloured bottle and store in a dark place.

A-5.3 Volumetric Method — Method B

A-5.3.1 *Reagents*

A-5.3.1.1 *Silver nitrate solution* **—** 0.01 N

Weigh accurately 0.169 9 g of silver nitrate (AR grade) and dissolve in chloride free water. Add 10 ml of concentrated nitric acid and dilute to 1 litre with water. Store the solution in a coloured bottle and store in a dark place.

A-5.3.1.2 *Potassium thiocyanate solution* — 0.01 N

Weigh accurately 0.971 7 g of potassium thiocyanate (AR grade), dissolve in water and dilute to 1 000 ml. Standardize this solution with standard 0.01 N silver nitrate solution.

A-5.3.1.3 *Ferric nitrate solution* **—** 40 percent

Weigh 40 g ferric nitrate (AR grade) and dissolve in a small quantity of water. Add a few drops of concentrated nitric acid (AR grade) and dilute to 100 ml.

A-5.3.1.4 *Potassium chloride solution* **—** 0.02 percent

Weigh accurately 0.2 g of potassium chloride (AR grade), dissolve in chloride free water and dilute to 1 000 ml.

A-5.3.1.5 *Concentrated nitric acid*

A-5.3.1.6 *Nitrobenzene*

A-5.3.2 *Procedure*

A-5.3.2.1 Take 10 ml of standard potassium chloride solution **A-5.3.1.4** and add about 50 ml of chloride free water. Add 5 ml of concentrated nitric acid and 2 ml of ferric nitrate solution **A-5.3.1.3**. Add 10 ml of 0.01 N silver nitrate solution followed by 2 ml of nitrobenzene, stir vigorously and titrate with 0.01 N potassium thiocyanate solution till the faint brick red colour appears. From the consumption of standard silver nitrate solution calculate the equivalent of potassium chloride for 1 ml of silver nitrate solution.

Let 1 ml of standard silver nitrate solution = X mg of potassium chloride

A-5.3.2.2 Weigh 10 g of potassium hydroxide sample and transfer to a 250 ml conical flask. Add a few millilitres of water and neutralize with concentrated nitric acid. Add 2 ml of ferric nitrate solution **A-5.3.1.3** and 10 ml of standard silver nitrate solution. Add 2 ml of nitrobenzene and, titrate against 0.01 N potassium thiocyanate solution till brick red colour appears.

A-5.3.3 *Calculation*

$$\text{Chloride (as KCl), ppm} = \frac{1\,000\,(10 - V) \times X}{M}$$

where

V = volume, in ml of standard potassium thiocyanate solution used in the titration;

X = mass, in mg, of potassium chloride equivalent to 1 ml of silver nitrate; and

M = mass; in g, of the material taken for the test.

A-5.4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed in **A-21**.

A-6 TEST FOR SULPHATES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50 ml capacity

A-6.2 Reagents

A-6.2.1 *Dilute Hydrochloride Acid* — 5 N

A-6.2.2 *Barium Chloride Solution* — approximately 10 percent (*m/v*)

A-6.2.3 *Standard Sulphate Solution*

Dissolve 0.181 4 g of potassium sulphate in water and dilute to 1 000 ml. One millilitre of this solution contain 0.1 mg of sulphate as SO_4 or 0.18 mg as K_2SO_4.

A-6.2.4 *Dilute Standard Sulphate Solution*

Dilute 10 ml of standard sulphate solution to 100 ml. One millilitre of diluted solution contains 0.01 mg of sulphate (as SO_4) or 0.018 mg as K_2SO_4.

A-6.3 Procedure

Transfer by means of a pipette 10 ml in case of pure and AR grades and 25 ml in case of ER grade, of the prepared sample solution (*see* **A-2**) in a Nessler cylinder, add 5 ml of dilute hydrochloric acid and 2 ml of barium chloride solution; mix with a glass rod and dilute to the mark. Carry out a control test in, another Nessler cylinder using 3 ml of dilute standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-6.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that in the control test.

A-6.4 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed in **A-21**.

A-7 TEST FOR PHOSPHATES

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50 ml capacity

A-7.2 Reagents

A-7.2.1 *Dilute Sulphuric Acid* — approximately 5 N

A-7.2.2 *Phosphate Reagent No. 1*

Dissolve 5 g of ammonium molybdate in 100 ml of 1 N sulphuric acid.

A-7.2.3 *Phosphate Reagent No. 2*

Dissolve 0.2 g of p-methyl aminophenol sulphate (metal) and 20 g of potassium metabisulphite in 100 ml of water.

A-7.2.4 *Standard Phosphate Solution*

Dissolve 1.43 g of potassium di-hydrogen phosphate in water and dilute to 1 000 ml. Store in a polyethylene, bottle. Dilute 10 ml of this solution to 1 000 ml in a measuring flask immediately before use. One millilitre of the diluted solution contains 0.01 (as PO_4).

A-7.3 Procedure

A-7.3.1 Dissolve 4.0 g of the material in 20 ml of water in a platinum dish, neutralize with dilute sulphuric acid, add 2 ml of the acid in excess and dilute to 50 ml.

A-7.3.2 Transfer 25 ml of the above solution into a Nessler cylinder. Add 2 ml of dilute sulphuric, acid, 1 ml of phosphate reagent No. 1, 1 ml of phosphate reagent No. 2 and dilute to 50 ml with water. Carry out a control test in another Nessler cylinder using 1 ml of standard phosphate solution and the same quantities of other reagents in the same total volume of the reaction mixture. Keep both the cylinders in a water-bath at 60 °C for 10 min and then compare the colour produced in both the cylinders after 5 min.

A-7.3.3 The limit prescribed for phosphate shall be taken as not having been exceeded if any blue colour developed in the test with the material is not deeper than that produced in the control test.

A-8 TEST FOR IRON

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinder* — 50 ml capacity

A-8.2 Reagents

A-8.2.1 *Concentrated Hydrochloric Acid* — (rd 1.16)

A-8.2.2 *Ammonium Persulphate*

A-8.2.3 *Butanolic Potassium Thiocyanate*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make-up to 100 ml and shake vigorously until the solution is clear.

A-8.2.4 *Dilute Sulphuric Acid* — approximately 5 N

A-8.2.5 *Standard Iron Solution*

Dissolve 0.702 g of ammonium ferrous sulphate in about 100 ml of water containing 10 ml of dilute sulphuric acid (5 N) and dilute to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-8.2.6 *Diluted Standard Iron Solution*

Further dilute 100 ml of the standard iron solution (**A-8.2.5**) to 1 000 ml. One millilitre of this diluted solution contains 0.001 mg of iron (as Fe).

A-8.3 Procedure

Transfer 10 ml of the prepared sample solution (*see* **A-2**) in a Nessler cylinder and make it acidic with hydrochloric acid. Add 1 g of ammonium persulphate, boil for one minute, cool and add 15 ml of butanolic potassium thiocynate. Make up the volume to 50 ml and shake vigorously and allow the liquid to separate. Carry out a control test using 2.5 ml of standard iron solution for the pure grade and 5 ml of dilute standard iron solution for the AR and ER grades of the material, and the same quantities of the other reagents in the same total volume of the reaction mixture.

A-8.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the red colour produced in the test with the material is not deeper than that produced in the control test.

A-8.4 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-9 TEST FOR HEAVY METALS

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* **—** 50 ml capacity

A-9.2 Reagents

A-9.2.1 *Dilute Hydrochloric Acid* **—** approximately 5 N

A-9.2.2 *Dilute Ammonium Hydroxide* **—** approximately 5 N

A-9.2.3 *Potassium Cyanide Solution*

Dissolve 10 g of potassium cyanide in 100 ml of water.

A-9.2.4 *Acetic Acid* — glacial

A-9.2.5 *Hydrogen Sulphide Solution* — saturated and freshly prepared solution in water

A-9.2.6 *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Dilute 10 ml of this solution to 1 000 ml with water. One millilitre of the diluted solution contains 0.01 mg of lead (as Pb).

A-9.3 Procedure

Dissolve 2 g of the material in water in a Nessler cylinder, add 5 ml of dilute hydrochloric acid, 10 ml of dilute ammonium hydroxide, 1 ml of potassium cyanide solution and dilute to about 40 ml. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test, using 2 ml of standard lead solution for pure grade and 1 ml of standard lead solution for the AR and ER grades. 1 ml of acetic acid and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in both the cylinders after 5 min.

A-9.3.1 The limit prescribed for heavy metals (as Pb) shall be taken as not having been exceeded if any colour produced in the test with the material is not darker than that produced in the control test.

A-9.4 Alternative Method

Lead may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-10 DETERMINATION OF INSOLUBLE MATTER

A-10.1 Procedure

Dissolve 50 g of the sample in 500 ml of carbon dioxide free water and heat on a steam bath for 30 min. Filter through a tared Gooch crucible, wash the residue with water till it is free from alkali and dry at 105 °C ± 2 °C. Collect the filtrate and washings in a 1 000 ml graduated flask and dilute to the mark.

A-10.2 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{100m}{M}$$

where

m = mass, in g, of the residue; and
M = mass, in g, of the material taken for the test.

A-11 DETERMINATION OF SODIUM

A-11.1 Apparatus

A-11.1.1 *Flame Photometer* — equipped with interference filter.

NOTE — Sodium filter to be used should be of preselected or metal filter type, since ordinary sodium filters (for example, gelatine filters) have only 1 percent selectivity and work properly when the ratio of Na : K is 100 : 1. Whereas in potassium carbonate and potassium hydroxide, the ratio is exactly reverse. Hence results with gelatine filter are erratic.

A-11.2 Reagent

A-11.2.1 *Standard Sodium Solution*

Weigh exactly 2.542 g of sodium chloride, dissolve in deionized water and dilute to 1 000 ml in a measuring flask. Dilute 10 ml of this solution to 1 000 ml. One ml of the diluted solution contains 0.01 mg of sodium (as Na).

A-11.3 Sample Solution

Dissolve exactly 10 g of the pure grade of material (20 g in case of AR grade and 200 g in case of ER grade) in deionized water and dilute to 1 000 ml in a volumetric flask. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. Use this solution for galvanometer readings. If needed, further dilute the solution.

A-11.4 Calibration Graph Take 10 ml, 20 ml, 40 ml, 60 ml and 80 ml of standard sodium solution in different measuring flasks and dilute to 100 ml with deionized water. Each flask now contains 0.1 mg, 0.2 mg, 0.4 mg, 0.6 mg and 0.8 mg of sodium per 100 ml. Use these diluted solutions to obtain corresponding galvanometer reading as given in the procedure (**A-11.5.2**) and plot the concentration against galvanometer reading. Draw a smooth curve over the points which gives a calibration graph in the range 0.1 mg to 1.0 mg of sodium per 100 ml.

A-11.5 Procedure

A-11.5.1 Use a flame photometer equipped with atomizer, burner, optical selective device consisting of reflectors, lenses and diaphragms, and measuring instrument consisting of a photocell, an amplifier and a sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

A-11.5.2 Insert the sodium filter corresponding to wavelength 767 nm light burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 kg/cm^2 to 0.7 kg/cm^2 and maintain the control knob. First spray deionized water and adjust the pointer to zero in galvanometer scale by adjustment knob. Then spray the standard sodium solution and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray deionized water to see that 'pointer comes to zero: then spray standard solution to indicate 100. Repeat till deionized water reads zero and standard solution reads 100 with the same adjustment during both the operations. Reading zero by deionized water and with the same adjustment 100 by standard solution, indicates that the instrument is now ready for measurement.

A-11.5.3 Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in **A-11.4** and obtain a calibration graph in the range 0.1 mg to 1.0 mg of sodium per 100 ml. After washing with deionized water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of sodium (say A).

A-11.6 Calculation

$$\text{Sodium (as Na), percent by mass} = \frac{10\text{ A}}{M}$$

where

A = concentration of sodium, in mg, per 100 ml corresponding to the galvanometer reading (*see* **A-11.5.3**); and
M = mass, in g, of the material taken for preparing the sample solution.

A-11.7 Alternative Method

Sodium may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-12 TEST FOR ARSENIC

A-12.1 Dissolve 1.000 g of the material in 10 ml of water and carry out test for arsenic as prescribed in IS 2088 using 0.001 32 mg of arsenic trioxide comparison stain. (As_2O_3) for preparing the comparison stain.

A-12.2 Alternative Method

Arsenic may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-13 TEST FOR NITRATES

A-13.1 Reagents

A-13.1.1 *Dilute Sulphuric Acid* — approximately 5 N

A-13.1.2 *Concentrated Hydrochloric Acid* — (*see* IS 265)

A-13.1.3 *Standard Potassium Nitrate Solution* — 0.001

A-13.1.4 *Concentrated Sulphuric Acid* — (*see* IS 266)

A-13.1.5 *Standard Indigo Carmine Solution*

Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid, add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to make up the volume to 1 000 ml. Dilute 100 ml of this solution to 1 000 ml. Standardize the solution so that 100 ml added to 3.3 ml of potassium nitrate solution is just decolourized on adding 13 ml of concentrated sulphuric acid and heat to boiling. One millilitre of this solution is equivalent to 0.002 mg of nitrate (as NO_3).

A-13.1.6 *Dilute Ammonium Hydroxide* — approximately 5 N

A-13.2 Procedure

Weigh accurately 2 g of the material and dissolve in 100 ml of water. Add 50 ml of dilute ammonium hydroxide and filter by suction. To 15 ml of the filtrate add 5 ml of dilute sulphuric acid, 1 ml of standard indigo carmine solution and 20 ml of concentrated sulphuric acid and heat to boiling.

A-13.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced does not entirely disappear.

A-13.3 Alternative Method

Nitrates may alternatively be determined by instrumental test method as prescribed in **A-21**.

A-14 TEST FOR SILICATES

A-14.1 Apparatus

A-14.1.1 *Nessler Cylinders* — 50 ml capacity

A-14.2 Reagent

A-14.2.1 *Dilute Sulphuric Acid* — approximately 1 N

A-14.2.2 *Ammonium Molybdate Solution*

Dissolve 10 g of ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

A-14.2.3 *Sodium Citrate Solution*

Dissolve 8 g of sodium citrate ($Na_3C_6H_5O_7.H_2O$) in 100 ml of water.

A-14.2.4 *Potassium Metabisulphite Solution*

Dissolve 20 g of potassium metabisulphite and 1 g of hydroquinone in 100 ml of water.

A-14.2.5 *Anhydrous Sodium Carbonate*

A-14.2.6 *Standard Silicate Solution*

Fuse 0.100 g of pure, precipitated silica with anhydrous sodium carbonate in a platinum crucible and leach the melt with water. Neutralize with dilute sulphuric acid and dilute to 1 000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of silica (as SiO_2). Store in a polyethylene bottle.

A-14.3 Procedure

Dissolve 2 g of water in a platinum the material in 20 ml of dish, neutralize with about 10 ml of dilute sulphuric acid, add 2 ml of the acid in excess and dilute to 50 ml. Transfer 10 ml of this solution into a Nessler cylinder. Add 1 ml of dilute sulphuric acid and 5 ml of ammonium molybdate solution. Allow to stand for 15 min and then add 5 ml of sodium citrate solution followed by 5 ml of potassium metabisulphite solution. Dilute to 50 ml. Carry out a control test in another Nessler cylinder using 8 ml of standard silicate solution in case of pure grade, 2 ml in case of AR grade and 1.6 ml in case of ER grade, in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Place both the cylinders in a boiling water bath for 5 min, cool and compare the colour produced in the two cylinders after 5 min.

A-14.3.1 The limit prescribed for silicates shall be taken as not having been exceeded if any blue colour produced in the test with the material is not deeper than that produced in the control test.

A-15 TEST FOR ALUMINIUM

A-15.1 Apparatus

A-15.1.1 *Nessler Cylinders* — 50 ml capacity

A-15.2 Reagents

A-15.2.1 *Dilute Acetic Acid*

Dissolve 28.5 g of glacial acetic acid in 100 ml of

water.

A-15.2.2 *Ammonium Aurine Tricarboxylate* (*Aluminon*) *Solution*

Dissolve 0.1 g of ammonium aurine tricarboxylate in 100 ml of water.

A-15.2.3 *Ammonium Carbonate Solution* — approximately 5 N

A-15.2.4 *Standard Aluminium Solution*

Dissolve 1.688 0 g of aluminium ammonium sulphate {$AINH_4$ $(SO_4)_2.12$ H_2O} and dilute to 1 000 ml. One millilitre of the diluted solution contain 0.1 mg aluminium (Al). Dilute 10 ml of this solution to 100 ml. One ml of this diluted solution contains 0.01 mg of aluminium (as Al).

A-15.2.5 *Ammonium Acetate — solid*

A-15.3 Procedure

Transfer 5 ml of the sample solution into a Nessler cylinder. Add 10 ml of dilute acetic acid and 1 ml of ammonium aurine tricarboxylate solution. Allow to stand for 5 min and then add 10 ml of ammonium carbonate solution. Carry out a control test in another Nessler cylinder using 1.0 ml of diluted standard aluminium solution in case of AR Grade and 0.5 ml in case of ER Grade, 15 ml of water, 1 g of ammonium acetate, 5 ml of dilute acetic acid, 1 ml of ammonium aurine tricarboxylate solution and adding, after a lapse of 5 min, 10 ml of ammonium carbonate solution. Make up the volume in the two cylinders to 50 ml and compare the colour produced in the two cylinders after 5 min.

A-15.3.1 The limit prescribed for aluminium shall be taken as not having been exceeded if any pink colour produced in the test with the material is not deeper than that produced in the control test.

A-15.4 Alternative Method

Aluminium may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-16 TEST FOR AMMONIUM SALTS

A-16.1 Reagents

A-16.1.1 *Nessler Solution*

Dissolve 143 g of sodium hydroxide in 700 ml of water. Dissolve 50 g of red mercuric iodide and 40 g of potassium iodide in 200 ml of water. Pour the iodide solution into hydroxide solution and dilute to 1 000 ml. Allow to settle and use the clear supernatant liquid.

A-16.1.2 *Standard Ammonium Chloride Solution*

Dissolve 0.296 g of ammonium chloride (NH_4Cl) in water and dilute to 100 ml. Dilute 1 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.001 mg of ammonium (as NH_4).

A-16.2 Procedure

Take 2 ml of the sample solution (*see* **A-2**), dilute with water to 50 ml and add 2 ml of Nessler solution. In a control test, having an equal volume of the solution and containing 1 ml of standard ammonium chloride solution, add 2 ml of Nessler solution.

A-16.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced with the material is not darker than that produced with the standard solution.

A-17 DETERMINATION OF CALCIUM GROUP AND MAGNESIUM

A-17.1 Reagents

A-17.1.1 *Standard Calcium Solution*

Weigh 1 g of calcium carbonate dried at 120 °C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a graduated flask. One millilitre of the solution is equivalent to 0.400 8 mg of calcium (as Ca).

A-17.1.2 *Standard EDTA Solution*

Dissolve 3.72 g of disodium ethylene diamine tetracetate dihydrate in water and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against the standard calcium solution following the procedure given in **A-17.2.1**.

A-17.1.3 *Eriochrome Black T Indicator Solution*

Dissolve 0.1 g of the dye in 20 ml of rectified spirit *see* IS 323. This solution shall be prepared fresh every week.

A-17.1.4 *Ammonium Chloride* — ammonium hydroxide buffer solution

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (rd 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetracetate dihydrate and 0.616 g of magnesium sulphate ($MgSO_4.7H_2O$) in about 50 ml of water. Mix the two solutions and dilute to 1 litre.

NOTE — Five millilitres of the buffer solution added to 50 ml of water should not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

A-17.2 Procedure

A-17.2.1 *Standardization of EDTA Solution*

Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution,5 drops of the eriochrome black T indicator and titrate against the standard EDTA solution to a clear blue end point.

A-17.2.2 Titrate 25 ml of the buffer solution with EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0.1 ml) from the reading obtained in **A-17.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (A).

A-17.2.3 Transfer exactly 100 ml of the sample (*see* **A-2**) solution into a 250 ml conical flask. Add 20 ml of ammonium chloride-ammonium hydroxide buffer solution and 5 drops of eriochrome black T indicator. Titrate against standard EDTA solution till wine red colour of the solution changes to clear blue end-point. Note the volume of EDTA solution used in the titration (v).

A-17.3 Calculation

Calcium group and magnesium (as Ca),

$$\text{percent by mass (on dry basis)} = \frac{A\,V_1}{M}$$

where

A = calcium equivalent, in mg, of 1 ml of EDTA solution determined in **A-17.2.2**;

V_1 = volume, in ml, of standard EDTA solution used in **A-17.2.3**; and

M = mass, in g, of the material in the aliquot taken for the test.

A-17.4 Alternative Method

Calcium and magnesium may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-18 TEST FOR NICKEL

A-18.1 Apparatus

A-18.1.1 *Nessler Cylinders* — 50 ml capacity

A-18.2 Reagents

A-18.2.1 *Citric Acid* — crystals

A-18.2.2 *Dilute Ammonium Hydroxide* (rd 0.90)

A-10.2.3 *Dimethyl Glyoxime Solution* — 1 percent (m/v) solution in alcohol

A-18.2.4 *Standard Nickel Solution*

Dissolve 6.727 g of nickel ammonium sulphate [$NiSO_4.(NH_4)2SO_4.6H_2O$] in water and dilute to make up the volume to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of nickel (as Ni).

A-18.3 Procedure

Transfer 5 ml of the prepared sample solution in a Nessler cylinder and dilute with water to 15 ml. Add 2 g of citric acid and dilute ammonia solution until the contents become slightly alkaline to litmus paper. Add 1 ml of dimethyl glyoxime and make up the volume to 50 ml with water. Carry out a control test, using 1 ml of standard nickel solution for pure grade and 0.5 ml for AR grade and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced after 5 min.

A-18.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if any pink colour produced is not deeper than that produced in the contro1 test.

A-18.4 Alternative Method

Nickel may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-19 TEST FOR ZINC

A-19.1 Apparatus

A-19.1.1 *Nessler Cylinders* — 50 ml capacity

A-19.2 Reagents

A-19.2.1 *Citric Acid* — crystals

A-19.2.2 *Dilute Ammonium Hydroxide* (rd 0.90)

A-19.2.3 *Resorcinol Solution* — approximately 5 N

A-19.2.4 *ThymoI Blue Indicator*

Warm 0.1 g of thymol blue with 4.3 ml of 0.05 N sodium hydroxide solution and 5 ml of rectified

spirit until dissolved. Dilute with ethyl alcohol (20 percent by volume).

A-19.2.5 *Chloroform*

A-19.2.6 *Dithizone Solution*

Dissolve 0.1 g diphenylthiocarbazone ($C_4H_5NHNHCSN{:}NC_6H_5$) in 100 ml of chloroform.

A-19.2.7 *Standard Hydrochloric Acid* — 1 N

A-19.2.8 *Ammonium Chloride* — solid

A-19.2.9 *Potassium Ferrocyanide Solution* — approximately 1 percent, freshly prepared solution in water

A-19.2.10 *Standard Zinc Solution*

Dissolve 1.0 g of zinc in slight excess of dilute hydrochloric acid and dilute with water to make up the volume to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of zinc (as Zn).

A-19.3 Procedure

Pipette 10 ml of the prepared sample solution (*see* **A-2 1** in a beaker and add 1 g of citric acid, 0.1 ml of resorcinol solution and adjust the *p*H of the solution to approximately 9.0 using dilute ammonium hydroxide and thymol blue indicator. Shake vigorously with three successive 5 ml portions of dithizone solution. Mix the chloroform extracts, wash with 5 ml of water and then shake with 10 ml of standard hydrochloric acid. Separate and wash the acid with 5 ml of chloroform. Transfer to a Nessler cylinder, add 1 g of ammonium chloride and dilute with water to about 45 ml. Make up the volume with water to 50 ml and add 1 ml of potassium ferrocyanide solution. Carry Out a control test in another Nessler cylinder using 1.5 ml of standard zinc Solution in place of the prepared sample solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidities after 15 min.

A-19.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-19.4 Alternative Method

Zinc may alternatively be determined by instrumental test method as prescribed in **A-20**.

A-20 DETERMINATION OF ALUMINIUM, ARSENIC, CALCIUM, IRON, LEAD, MAGNESIUM, NICKEL, SODIUM AND ZINC BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-20.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency Inductively Coupled Plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-20.2 Recommended Wavelength, Limit of Quantification and Important Spectral Interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

A-20.3 Reagents and Solutions

A-20.3.1 *Nitric Acid* (*65 percent*) *Suprapure*

A-20.3.2 *Standard Stock Solution*

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10 µg/ml, 100 µg/ml or 1 000 µg/ml of aluminium, arsenic, calcium, iron, lead, magnesium, nickel sodium and zinc in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clause* A-20.2)

Sl No.	**Element**	**Wavelength** (nm)	**Approximately Achievable Limits**		**Interfering Elements**
			Radial Viewing (µg)	Axial Viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)	(20)	Cr, Fe
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	—	—	—
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	—	—	V, Mo, Zr
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr
v)	Na	330.237	20	300	Zn
		588.995	20	200	Ar, V
		589.592	93	20	Ba
vi)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	100	31	Al, Co, Fe, Pb, Ti
vii)	Ni	221.648	10	2	Si
		231.604	15	2	Co, Sb
viii)	Zn	202.548	—	3	Cr, Cu, Co, Ni
		206.200	13	5	Cr
		213.857	3.3	1	Cu, Fe, Ni
ix)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr

A-20.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 µg/ml, 0.3 µg/ml and 0.5 µg/ml solution of respective elements under reference.

A- 20.3.4 *Sample Preparation*

Weigh about 2.5 g sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

A-20.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-20.4 Instrument

Set up the instrument as per the manufacturer’s instructions manual for recommended operating parameters, based on the manufacturers operating

manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

A-20.5 Procedure

A-20.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions **A-20.3.3**. The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

A-20.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution **A-20.3.5** between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the Aluminium, arsenic, calcium, iron, lead, magnesium, nickel sodium and zinc in the sample solution.

NOTE — It is recommended that IS 3025(Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

A-20.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

a) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot; and

b) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (dilution factor).

A-21 ION CHROMATOGRAPHY FOR CHLORIDES, NITRATES AND SULPHATES

A-21.1 Principle

Ion chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the *p*H of the column. Similarly in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-21.2 Equipment

A-21.2.1 *Anion Guard Column* — A protector of the separator column.

A-21.2.2 *Anion Separator Column* — Suitable for selective separation of ions under analysis.

A-21.2.3 *Anion Suppressor Device* — Anion micromembrane suppressor is used to analyse the data.

A-21.2.4 *Detector* — Conductivity detector.

A-21.2.5 *Software*

Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-21.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-21.3 Reagents

A-21.3.1 *Glass or Polyethylene Sample Bottles*

A-21.3.2 *Distilled Water or Deionized Water free from the Anions of Interest*

A-21.3.3 *Eluent*

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litre of water.

A-21.3.4 *Micromembrane Suppressor Solution*, (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentarted Sulphuric acid in 4 litre of water.

A-21.4 Standard Solutions

A-21.4.1 *Chloride*

Dissolve NaCl, 1.648 5 g in 1 litre of reagent water.

A-21.4.2 *Sulphate*

Dissolve 1.81 g of potassium sulphate in 1 litre of reagent water.

A-21.4.3 *Nitrate Stock Solution*

Dry approximately 2 g of sodium nitrate ($NaNO_3$) at 105 °C for 24 h. Dissolve exactly 1.370 7 g of the dried salt in reagent water, and dilute to 1 litre with reagent water in a volumetric flask.

A-21.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-21.6 Procedure

Dissolve between 1 g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-21.7 Data Analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

ANNEX B

(*Clause* 6)

SAMPLING OF CAUSTIC POTASH

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Precautions shall be taken to protect the sample and sample containers from any possible contamination.

B-1.2 Utmost care shall be taken in handling the material in the course of taking out and transferring the sample.

B-1.3 From each of the selected container, representative sample shall be taken after thoroughly mixing the contents.

B-1.4 These samples shall be placed in clean, dry, tight and alkali, resistant glass containers.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be identified and separated to form respective lots.

B-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. The number (n) of containers to be selected at random from lot of size (N) shall be in accordance with Table 3.

B-2.3 These containers shall be drawn at random from the lot. To ensure randomness, random number tables may be used as indicated in IS 4905.

B-3 TEST SAMPLES

B-3.1 Draw with an appropriate sampling implement small portion of caustic potash from different parts of the container. The total amount collected from each of the container shall be about 1.5 kg. These individual samples of l kg to 5 kg each shall be transferred to separate sample containers. From each of the individual samples equal amount of material shall be taken and mixed together to get a composite sample of mass 2.0 kg. The composite sample shall then be divided into three identical parts, one for the purchaser, another for the supplier and the third to be kept as referee sample.

B-3.2 Test for total alkalinity (as KOH) shall be conducted on each of the individual samples. Tests for other characteristics shall be performed on the composite sample.

B-4 CRITERIA FOR CONFORMITY

B-4.1 Total Alkalinity

The individual test results for total alkalinity shall be noted. From these results, mean ($\bar{X}$) and the range (R), that is, the difference between the maximum and of values shall be computed. The lots shall be accepted in respect of total alkalinity only if ($\bar{X}$ - 0.4 R) is greater than or equal to 87.0.

B-4.2 For all other characteristics the total results obtained on the composite sample shall satisfy the corresponding requirements if the lot is to be deemed acceptable.

Table 3 Scale of Sampling

(*Clause* B-2.2)

Sl No.	Lot Size	Number of Containers to be Selected
	(N)	(n)
(1)	(2)	(3)
i)	up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 300	6
v)	301 and above	8

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR KANNAN SRINIVASAN **(*Chairperson*)**
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